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Indian Standard

SPECIFICATION FOR DOUBLE-TEXTURE RUBBERISED WATERPROOF FABRICS

(First Revision)

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INDIAN STANDARDS INSTITUTION
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Indian Standard

SPECIFICATION FOR DOUBLE-TEXTURE RUBBERISED WATERPROOF FABRICS

(Fourth Revision)

Treated Fabrics Sectional Committee, PCDC 16

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Indian Standard

SPECIFICATION FOR DOUBLE-TEXTURE RUBBERISED WATERPROOF FABRICS

(First Revision)

0. FOREWORD

- **0.1** This Indian Standard was adopted by the Indian Standards Institution on 15 February 1983, after the draft finalized by the Treated Fabrics Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.
- 0.2 This standard was published in 1971 and covered only one quality of the material with not less than 55 percent rubber polymer content on the mass of the proofing. In this revision a second quality with rubber polymer content of the proofing with not less than 35 percent on the mass of the proofing has been added. Further each quality will now be in three grades depending on total mass, proofing content and the breaking strength of the rubberized fabric.
- **0.3** Double-texture rubberized fabrics are made by sandwiching a rubber composition between two sheets of base fabric. It is a heavy-duty cloth for use in the fabrication of ground sheets, waterproof garments, travelling bags, holdalls, etc. It may also be used as waterproof cover for general purposes.
- **0.4** This standard contains clause **5.2.4** which calls for an agreement between the purchaser and the supplier.
- **0.5** For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS: 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

^{*}Rules for rounding off numerical values (revised).

1. SCOPE

1.1 This standard prescribes the requirements and the methods of test for double texture rubberized waterproof fabrics used for defence and general purposes.

2. DESCRIPTION

2.1 The double-texture rubberized waterproof fabrics shall consist of two plies of dyed and/or undyed cotton fabric in viscose as required by the purchaser and laminated longitudinally with a thin intervening layer of rubber proofing.

3. QUALITIES AND GRADES

- **3.1 Qualities** The double texture rubbrized waterproof fabrics shall be of two qualities based on the minimum rubber polymer content of the proofing by mass as indicated in **3.1.1** and **3.1.2**.
- 3.1.1 Quality 1 Minimum rubber polymer content of the proofing shall be 55 percent by mass, when tested according to Appendix A.
- 3.1.2 Quality 2 Minimum rubber polymer content of the proofing shall be 35 percent by mass, when tested according to Appendix A.

Note — The direct method for determination of rubber polymer content given in Appendix B may be used when the manufacturer has declared that he has used only natural rubber composition for proofing.

3.2 Grades — The double texture rubberized waterproof fabrics of each quality shall be of three grades as described in Table 1.

4. WORKMANSHIP AND FINISH

4.1 The double texture rubberized waterproof fabrics shall be flexible and shall have the two cloth layers firmly adhered to the proofing. It shall be free from bare places, rubber patches on the surface, oil and other stains which are liable to effect the serviceability of the finished fabric. It shall also be reasonably free from creases, wrinkles, thin places, uneven shade, pin holes and other manufacturing defects.

5. REQUIREMENTS

5.1 Base Fabric — The base fabric for the manufacture of double texture rubberized waterproof fabric shall be made of cotton or viscose staple or other suitable textile material as agreed to beetween the purchaser and the supplier.

5.2 Rubber Proofing

- 5.2.1 The proofing shall be made from natural rubber or suitable vulcanizable synthetic rubber or a combination thereof compounded with the necessary ingredients.
- 5.2.2 It shall not contain substances, such as copper and manganese compounds in such amounts as to have deleterious action on rubber. Reclaimed rubber may be used in quality 2.
- 5.2.3 The proofing shall be non-irritant and free from objectionable odour.
- **5.2.4** Resistance to Cold The proofing within the double-texture rubberized fabric shall withstand a bending test around a steel pin of 6 mm diameter without cracking, immediately after an exposure of 5 hours to a temperature of minus $35 \pm 1^{\circ}\mathrm{C}$. This requirement is optional and shall be agreed to between the purchaser and the supplier.
- **5.2.5** Acetone Extract and Extractable Sulphur The amounts of acetone extract and extractable sulphur, as determined in Appendix C shall not exceed the following limits (percent by mass):

	Cotton Fabrics	Other Fabrics
Acetone extract Extractable sulphur	8 0 0·5	15·0 0·5

5.3 Finished Fabrics

5.3.1 General Requirements

5.3.1.1 Length — Unless otherwise specified, the finished fabric shall be supplied in minimum length of 30 m without any joints. Acceptance of the fabrics in length of less than 30 m or with joints for length over 30 m shall be as agreed between the purchaser and the supplier.

 ${
m Note}$ — Maximum of 5 percent of the supply may be made in short length, provided no individual length is less than 10 m.

- **5.3.1.2** Width The minimum effective width of the finished fabrics shall be 115 cm or as agreed to between the purchaser and the supplier.
- 5.3.1.3 The finished fabrics shall also comply with the requirements prescribed in Table 1 when tested according to the method indicated in col 9 of Table 1.
- **5.3.2** Waterproofness Test The finished fabrics shall hold a 90 cm head of water for one hour without allowing any percolation of water when tested in accordance with Appendix E.

5.3.3 Accelerated Ageing — The finished fabric, after being subjected to accelerated again at a temperature of $90 \pm 1^{\circ}\text{C}$ for 150 hours in an airoven, shall show no sign of softening, stiffening or other apparent deterioration of rubber and shall satisfy the test specified under **5.3.2**.

TABLE 1 REQUIREMENTS FOR FINISHED FABRIC

(Clauses 3.2 and 5.3.1.3)

SL No.	CHARACTERISTIC	QUALITY 1		QUALITY 2			TEST METHOD, REF TO	
1101		Grade A	Grade B	Grade C	Grade A	Grade B	Grade C	
(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9)
i)	Mass*, g/m², <i>Max</i>	5 75	475	375	625	500	4 00	4.2 of IS: 7016 (Part I)-1982†
ii)	Proofing content, g/m², Min	250	200	150	300	2 50	200	Appendix D
iii)	iii) Breaking strength [‡] , kg/5 cm width, Min							IS: 7016 (Part II)-1981§
	Warp	65	45	50	65	45	50	
	Weft	30	30	40	30	3 0	4 0	

^{*}The mass shall be determined after conditioning the fabric for 48 h in an atmosphere of 65 ± 5 percent relative humidity and a temperature of $27 \pm 2^{\circ}$ C.

†Methods of test for coated and treated fabrics: Part I Determination of roll

characteristics (first revision).

\$1 kg = 9.8 N.

§Methods of test for coated and treated fabrics: Part II Determination of breaking strength and extension at break (first revision).

5.3.4 Adhesion of Plies — The average load required to strip off longitudinally either fabric ply from a test piece of the double-texture rubberized fabric 2.5 cm wide shall be not less than the following limits when tested in accordance with IS: 3400 (Part V)-1965*:

a) Original

1.5 kg

b) After accelerated ageing at 90 ± 1°C for 150 hours in air-oven

Not less than 70 percent of the original load

At least 4 test pieces cut from the sample, each in the direction of the warp and in the direction of the west shall be subjected to this test in original condition. A similar set of test pieces shall be subjected to this test after accelerated ageing.

^{*}Methods of test for vulcanized rubbers: Part V Adhesion of rubbers to textile fabrics.

5.3.5 Resistance to Xylole — When pieces of about 4 cm^2 of the finished fabric are immersed in xylole (see IS: 359-1965*) for a period of 2 hours and gently shaken in it for one minute, the plies shall not separate and the proofing shall not show any sign of tackiness or disintegration. The temperature of xylole, throughout the duration of test, shall be maintained at $27 + 2^{\circ}\text{C}$.

Note — Except where otherwise specified, the ambient air, throughout the duration of the above tests and for a period of 24 hours immediately preceding such tests, shall be maintained at a temperature of $27 \pm 2^{\circ}\text{C}$ and a relative humidity of 65 ± 5 percent.

- **5.3.6** Colour The finished material shall be of a suitable colour or different colour on the two sides as agreed to between the purchaser and the supplier. Sulphur black shall not be used. The colour fastness in respect of the coloured material shall comply with the following requirements.
- 5.3.6.1 Fastness to light The material when tested for colour fastness as prescribed in IS: 2454-1967† shall show a fastness rating of not less than No. 4 prescribed therein. In case of materials coloured on both sides or in dual shade, the test shall be conducted with respect to each side of the material separately and the colour fastness in each case assessed for compliance with the aforesaid requirement. The test shall be applicable to coloured as well as white rubberized fabrics.
- 5.3.6.2 Fastness to washing When the rubberized fabric is tested for colour fastness to washing in accordance with IS: 3361-1979‡, the fastness range, in respect of change in shade of the material and the degree of staining on the attached undyed pieces of cotton and clothes, evaluated according to IS: 768-1956§ and IS: 769-1956¶ respectively shall be not less than 5 in case of double texture rubberized fabrics where the base fabric is not dyed. In case of coloured fabrics where the base fabric is of dyed material, the fastness rating of the shades on each side shall be as agreed to between the purchaser and the supplier.
- **5.3.6.3** Reaction of aqueous extract When the finished fabric is tested in accordance with the 'hot method' of IS: 1390-1961¶, its aqueous extract shall neither be acidic to methyl orange nor alkaline to phenolphthalein indicator.

^{*}Specification for xylole, industrial solvent grade (revised).

[†]Method for determination of colour fastness of textile materials to artifical light (Xenon lamp).

[†]Method for determination of colour fastness of textile materials to washing: test 2 (first revision).

[§]Method for evaluating change in colour.

Method for evaluating staining.

Methods for determination of pH value of aqueous extracts of textile materials.

6. PACKING AND MARKING

- **6.1** The material shall be securely packed in rolls to ensure safe transportation.
- **6.2** Each roll shall be indelibly and legibly marked at both ends with the following:
 - a) Manufacturer's name;
 - b) Trade-mark, if any;
 - c) Month and year of manufacture;
 - d) Length of the piece in metres; and
 - e) Quality and grade of the material.
- **6.3** Other identification letters shall also be stamped on the material as required by the purchaser.
- 6.4 The material shall also be marked with the ISI Certification Mark.

Note — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

7. SAMPLING

7.1 The scale of sampling and the criteria for conformity of the material shall be as prescribed in Appendix F.

APPENDIX A

(Clauses 3.1.1 and 3.1.2)

DETERMINATION OF RUBBER POLYMER CONTENT (INDIRECT METHOD)

A-0. GENERAL

A-0.1 The indirect method determines the non-rubber constituents of a product and the remainder is expressed as rubber polymer content. The non-rubber constituents are determined in accordance with **A-1**

through A-9 and rubber polymer content is calculated as given in A-10. The method is applicable to NR, IR, SBR and BR products and also to IIR products if they are extracted with methyl ethyl ketone rather than with acetone.

Note — The abbreviations NR, IR, SBR, IIR, BR, CR, ABR and NBR used above and hereafter denote, isoprene (natural), isoprene (synthetic), styrene-butadiene, isobutylene-isoprene, butadiene, chloroprene, acrylate-butadiene and nitrite-butadiene rubbers.

A-1. TOTAL EXTRACT

A-1.1 Apparatus

A-1.1.1 Extraction Apparatus

A-1.1.2 Reagents

A-1.1.2.1 Acetone — distilled over potassium carbonate and boiling between 56 to 57°C (see IS: 170-1976*).

A-1.1.2.2 Chloroform

A-1.2 Procedure

- **A-1.2.1** Place a weighed specimen of approximately 2 g in a filter paper. If the specimen is in the form of a sheet, cut it with scissors into strips 3 to 5 mm in width. If the specimen becomes tacky during the extraction, take care that adjacent portions are separated by paper. Fold the paper so that it will fit in the extraction cup and suspend the cup in a weighed extraction flask containing 50 to 75 ml of a mixture consisting of 32 parts of acetone and 68 parts of chloroform by volume. (Prior to the weighing of the extraction flask, it shall have been dried for 2 hours at $70 \pm 5^{\circ}\mathrm{C}$ and cooled in a desiccator to the temperature of the balance.)
- A-1.2.2 Extract the specimen continuously for 16 hours, heating at a rate such that the time required to fill and empty the siphon cup shall be between 2.5 and 3.5 minutes. (Rubber products having a ratio of total sulphur to rubber polymer in excess of 10 percent shall be extracted for 72 hours.) Carefully note all characteristics of the extract, both when hot and cold. If the colour is black, make a chloroform extraction separately for 4 hours, dry the extract to constant weight and add the value to the result obtained for total extract.
- A-1.2.3 Evaporate off the solvent over a steam-bath, using a gentle current of filtered air to prevent boiling. Remove the flask from the steam-bath just prior to the disappearance of the last traces of solvent to

^{*}Specification for acetone (second revision).

prevent loss of extract. Continue the passage of air through the flask for 10 minutes to remove the remaining solvent and dry the flask for 2 hours in a 70 \pm 5°C air-bath.

A-1.2.4 Cool in a desiccator to the temperature of the balance and weigh. Save the extracted rubber for further tests that require the use of an extracted specimen.

A-1.3 Calculation

Total extract, percent by mass
$$= \frac{A}{B} \times 100$$

where

A = mass in g of extract, and

B =mass in g of specimen used.

A-2. ALCOHOLIC POTASH EXTRACT

A-2.1 Reagent

A-2.1.1 Alcoholic Potash Solution — Prepare 1 N alcoholic potassium hydroxide (KOH) solution by dissolving the required amount of potassium hydroxide in absolute ethyl alcohol that has been purified as follows:

Dissolve 1.5 g of silver nitrate (AgNO₃) in 3 ml of water and add it to 1 litre of alcohol. Dissolve 3 g of KOH in the smallest amount of hot water, cool, add it to the AgNO₃ solution and shake thoroughly. Allow the solution to stand for at least 24 hours, filter and distill.

Note — Absolute ethyl alcohol denatured with 10 percent by volume of methyl alcohol may also be used.

A-2.1.2 Congo Red Paper

A-2.1.3 Ethyl Alcohol - 95 percent.

A-2.1.4 Ether

A-2.2 Procedure

- **A-2.2.1** Remove the specimen remaining after the determination of total extract from its wrapping material while wet with solvent and dry the rubber at 70 ± 5 °C to remove the solvent.
- A-2.2.2 Transfer to a 200-ml Erlenmeyer flask, add 50 ml of alcoholic potash solution and heat under a reflux condenser for 4 hours. In the case of hard rubber, continue the heating for 16 hours or more.

- A-2.2.3 Filter into a 250-ml beaker, wash with two 25-ml portions of boiling alcohol and then with three 25-ml portions of boiling water and evaporate the filtrate just to dryness. Use about 75 ml of water to transfer the dried filtrate residue to a separatory funnel. Acidify the solution with hydrochloric acid (1:3) testing with Congo red paper.
- A-2.2.4 Extract with four 25-ml portions of other, unless the fourth portion should be coloured, when the extraction shall be continued until no further quantity can be removed. Unite the ether fractions and wash thoroughly with water until free of acid (two washings are generally sufficient).
- **A-2.2.5** Filter the ether solution through a plug of previously washed absorbent cotton into a weighed flask and wash the separatory funnel and the cotton plug with ether. Evaporate the ether on a steam-bath, using a gentle current of filtered air to prevent boiling. Remove the flask from the steam-bath just prior to the disappearance of the last traces of solvent and continue the passage of air for 10 minutes. Dry the flask at $100 \pm 5^{\circ}$ C to constant weight, cool and weigh.

A-2.3 Calculation

Alcoholic potash extract, percent = $\frac{A}{B} \times 100$

where

A = mass in g of extract, and

B =mass in g of specimen used.

A-3. TOTAL SULPHUR (ZINC-NITRIC ACID METHOD)

A-3.0 General — This method covers the determination of all the sulphur except that contained in barium sulphate (BaSO₄), in a sample of a rubber product or in the fillers obtained from a rubber product. If acid-soluble barium salts, antimony sulphide, or lead compounds are present this method will give erroneous results, in which case the fusion method (see A-4) should be used. This method may be used for the determination of organic plus inorganic sulphur on an extracted sample, total plus inorganic sulphur on an unextracted sample, or inorganic sulphur in filler. If it is used for determination of total organic sulphur plus inorganic sulphur, it shall also be used for determination of inorganic sulphur (see A-5). The method is applicable to NR, SBR, BR, IR and CR products and to total plus inorganic sulphur determination of NBR products.

A-3.1 Reagents

A-3.1.1 Barium Chloride Solution (BaCl₂.2H₂O), 100 g/l — Dissolve 100 g of barium chloride (BaCl₂.2H₂O) in water, add two to three

drops of hydrochloric acid and dilute to 1 litre. If there is any insoluble matter or cloudiness, heat the solution overnight on a steam-bath and filter.

- A-3.1.2 Bromine, Saturated Water Solution
- A-3.1.3 Nitric Acid, Fuming
- A-3.1.4 Picric Acid, Saturated Solution
- A-3.1.5 Potassium Chlorate (KClO3)
- A-3.1.6 Zinc-Nitric Acid Solution

A-3.2 Procedure

- A-3.2.1 Place 0.5 g of soft rubber or 0.2 g of hard rubber (extract the specimen with acetone or acetone-chloroform mixture if organic sulphur is to be determined) in a 500-ml Erlenmeyer distillation flask of chemically-resistant material. Add 10 ml of zinc-nitric acid solution and moisten the sample thoroughly. Let it stand for at least one hour, overnight if convenient. By so doing the sample becomes partly decomposed. This permits the addition of fuming nitric acid with no danger of ignition of the sample. Add 15 ml of fuming nitric acid and whirl the flask rapidly to keep the sample immersed to avoid ignition. With some sample it may be necessary to cool the flask under running water.
- **A-3.2.2** When the dissolution of the rubber appears to be complete, add 5 ml of a saturated water solution of bromine and slowly evaporate the mixture to a foamy syrup.
- A-3.2.3 If organic matter or carbon remains at this point, add a few millilitres of fuming nitric acid and a few crystals of potassium chlorate (caution) and evaporate at a boil. Repeat this operation until all carbon is removed and the solution is clear, colourless or light yellow.
 - A-3.2.4 At this point either of the following procedures may be used:

Procedure A — Place the flask on an asbestos gauze and evaporate the mixture to dryness over a burner. Then bake the mixture at the highest temperature of the burner until all nitrates are decomposed and no more nitrogen oxide fumes can be detected. The flask and its contents shall be carefully annealed after this procedure by gradually decreasing the flame or by placing the flask on successively cooler sources of heat.

Procedure B — Evaporate the mixture, cool, add 10 ml of hydrochloric acid and evaporate to dryness, avoiding spattering. Repeat this procedure once (or more) if oxides of nitrogen are still evolved.

A-3.2.5 Cool the flask, add 50 ml of hydrochloric acid (1:6) and digest hot until dissolution is as complete as possible. Filter while hot. Wash the filter and dilute the filtrate and washings to about 300 ml. Add 10 ml of saturated picric acid solution, heat to 90°C and precipitate the sulphate by dropwise addition of barium chloride solution while stirring vigorously. Digest the precipitate overnight, preferably at 60 to 80°C, using a watch glass to cover the beaker. Filter the barium chloride and wash with water until the filter is colourless. Dry, ash and finally ignite the precipitate at 650 to 900°C, with free access to air, to constant weight. Cool in a desiccator and weigh.

A-3.2.6 Calculation

Sulphur, percent by mass =
$$\frac{A \times 0.1373}{B} \times 100$$

where

A =mass in g of barium sulphate, and

B =mass in g of specimen used.

A-4. TOTAL SULPHUR (FUSION METHOD)

A-4.1 General — This method covers the determination of organic, total or inorganic sulphur in rubber compounds when acid-soluble barium salts, antimony sulphide or lead compounds are present. All of the sulphur in the sample including that present in barium sulphate is determined when this method is used. When this method is used for determination of organic or total sulphur it shall also be used for the determination of inorganic sulphur (see A-5). Total plus inorganic sulphur shall be determined on an unextracted specimen while organic plus inorganic sulphur on an extracted specimen. The method is applicable to NR, SBR, BR, IR and CR products and to the determination of total plus inorganic sulphur on NBR products.

A-4.2 Reagents

A-4.2.1 Nitric Acid-Bromine Solution — Add a considerable excess of bromine to nitric acid so that a layer of bromine is present in the reagent bottle. Shake thoroughly and allow to stand for 24 hours before using.

A-4.2.3 Sodium Carbonate Solution - 50 g/l.

A-4.3 Procedure

A-4.3.1 Place 0.5 g of soft rubber in a porcelain crucible of about 75 ml capacity. The specimen shall have been extracted with acetone

chloroform mixture (see A-1) of organic plus inorganic sulphur is to be determined. Add 15 ml of the nitric acid-bromine solution mixture, cover the crucible with a watch glass and let it stand for one hour in the cold. Heat for one hour on the steam bath, remove the cover, rinse it with a little water and evaporate to dryness.

- A-4.3.2 Add 3 ml of nitric acid, warm for a short time on the steambath, then allow to cool. Carefully add in small portions, by means of a glass spatula, 5 g of sodium carbonate. Raise the watch glass only high enough to permit the introduction of the spatula. Allow the sodium carbonate to slide down the side of the crucible as it shall not be dropped directly into the acid. Rinse the watch glass with 2 or 3 ml of hot water and stir the mixture thoroughly with a glass rod. Digest for a few minutes, spread the mixture half way up the side of the crucible to facilitate drying and dry on a steam-bath. Fuse the mixture by heating over a sulphur-free flame.
- A-4.3.3 Place the crucible in an inclined position on a wire triangle and start the ignition over a low flame. The tendency for the organic matter to burn too briskly may be controlled by judicious use of the stirring rod with which the burning portion is scraped away from the rest. When part of the mass is burned white, work a fresh portion into it until all of the organic matter is destroyed. It is necessary to hold the edge of the crucible with tongs. Towards the last half of the operation the flame should be increased. It is necessary to heat the crucible to redness.
- A-4.3.4 After a fusion, allow the crucible to cool. Place it in a 400-ml beaker, add sufficient water to cover the crucible (about 125 ml) and digest on the steam-bath or plate for at least two hours.
- A-4.3.5 Filter the solution into a covered 400-ml beaker containing 5 ml of hydrochloric acid and wash the residue thoroughly with hot sodium carbonate solution. A qualitative test for barium may be made on the residue, but no analysis for barium or correction because of its presence is necessary, unless a detailed ash analysis is desired. Acidify the filtrate to indicator paper with hydrochloric acid and add 2 ml in excess. Precipitate barium sulphate and complete the determination as described in A-3.

A-4.4 Calculation

Sulphur, percent by mass =
$$\frac{A \times 0.137 \text{ 3}}{B} \times 100$$

where

A =mass in grams of barium sulphate, and B =mass in grams of specimen used.

A-5. INORGANIC SULPHUR

A-5.1 General Type — This method covers the determination of inorganic sulphur in rubber products when no antimony is present. The sample is ashed and sulphur determined in the ash by the fusion method (see A-4) if acid-soluble barium salts or lead are present, or by the zinc-nitric acid method in the absence of acid-soluble barium salts. The same method shall be used for the inorganic sulphur determination as is used for the determination of organic sulphur or total sulphur. The method is applicable to NR, SBR, BR, IR, CR, NBR and IIR products.

A-5.2 Reagents — nitric acid-bromine solution.

A-5.3 Procedure — Extract 1.0 g specimen with acetone or with acetone-chloroform mixture (see A-1). Dry the sample, place in a porcelain crucible of about 75 ml capacity and distill off the rubber in a muffle furnace, using a maximum temperature of 450°C. A burner may be used for ashing if the sample is not allowed to catch fire. A wire gauze under the crucible will aid in preventing combustion. The carbon need not be completely burned off in this ignition. If acid-soluble barium salts and lead are absent, add 3 ml of nitric acid-bromine solution to the ash, cover with a watch glass and heat for one hour. Transfer the contents of the crucible, with washing, into a 500-ml Erlenmeyer destruction flask of chemically resistant material and evaporate to dryness. Proceed with the determination of sulphur as described in A-3.2.3 to A-3.2.6. In the presence of acid soluble barium salts and lead, determine the sulphur by treating the ash in accordance with A-1.5.

A-5.4 Calculation

Inorganic sulphur, percent by mass = $\frac{A \times 0.137 \text{ 3}}{B} \times 100$

where

A =mass in g of barium sulphate, and

B =mass in g of specimen used.

A-6. ORGANIC SULPHUR

A-6.1 Organic sulphur is calculated by subtracting inorganic sulphur as obtained in A-5 from total sulphur as obtained in A-3 or A-4 as applicable.

A-7. FILLERS, REFEREE ASH METHOD

A-7.1 General — This method is intended for settling disagreements on fillers content by the ashing method. It is also useful for ashing rubber

products for determination of inorganic sulphur in the absence of antimony or for ash analysis.

- A-7.1.1 The method is not accurate for rubbers containing halogens when zinc compounds or other metal compounds that form volatile halides are present and shall not be used if an analysis of these metals in the ash is required.
- A-7.1.2 The method may be used for preparing samples for ash analysis on rubber products not containing halogens or antimony sulphide. However, if the sample contains carbonates that decompose at 550°C or clays or silicates that will lose water at this temperature, the ash content value will not be highly precise and will not represent the original amount of inorganic fillers present in the sample.

A-7.2 Apparatus

- A-7.2.1 Crucible porcelain or silica, approximately 50 ml capacity.
- **A-7.2.2** Electric Muffle Furnace with controls necessary to hole the temperature at 550 ± 25 °C.
 - A-7.2.3 Calibrated Thermocouple and Temperature Readout Device

A-7.3 Procedure

- A-7.3.1 Weigh a 1-g specimen of the sample into an ignited, weighed crucible.
- **A-7.3.2** Adjust the temperature of the muffle furnace to $550 \pm 25^{\circ}$ C, place the crucible in the furnace and close completely. When more than one crucible is to be placed in the furnace, the crucible shall be placed on a tray and put into the furnace. The door shall be closed immediately and not opened for one hour. After one hour, open the furnace door 3 to 5 cm and continue heating for 30 minutes or until all carbonaceous material is burned off.
 - Note If a referee ash determination is required, the temperature shall be determined by placing the calibrated thermocouple sensing element at approximately the geometric centre of the furnace cavity. The temperature shall be adjusted to be within the specified range. Not more than two crucibles shall be placed in the oven and they shall be positioned directly below the thermocouple.
- A-7.3.3 Remove the crucible from the furnace, cool in a desiccator and weigh.

A-7.4 Calculation

Ash, percent by mass =
$$\frac{A-B}{C} \times 100$$

where

A =mass in g of ash and crucible,

B =mass in g of crucible, and

C =mass in g of specimen.

A-8. CARBON BLACK

A-8.1 General

A-8.1.1 This method covers the determination of carbon black by a nitric acid digestion method. It is applicable only to the 'R' family of rubbers containing an unsaturated carbon chain.

A-8.1.2 Application to any other rubber type may be possible if the rubber is degraded to fragments soluble in water, acctone, or chloroform. The method shall be used on such other rubbers only if adequate testing of known compounds has demonstrated the usefulness of the method.

A-8.2 Reagents

A-8.2.1 Acetone

A-8.2.2 Chloroform

A-8.2.3 Sodium Chromate (Na₂CrO₄) Solution — 100 g/l.

A-8.2.4 Sodium Hydroxide (NaOH) Solution - 175 g/l.

A-8.2.5 Sodium Hydroxide (NaOH) Solution - 300 g/l.

A-8.3 Procedure

A-8.3.1 Extract a 0.5 g specimen with acetone-chloroform mixture in accordance with **A-1**.

A-8.3.2 Transfer the specimen to a 250-ml beaker and heat on the steam-bath until it no longer smells of chloroform. Add a few millilitres of nitric acid and allow to stand for about 10 minutes. Add 50 ml more of nitric acid taking care to wash down the sides of the beaker and heat on the steam-bath for at least one hour. At the end of this time there should be no more bubbles or foam on the surface. Pour the liquid, while hot, into a Gooch crucible taking care to keep as much as possible of the insoluble material in the beaker. Filter slowly with gentle suction

and wash well by decantation with hot nitric acid [Caution — Empty the filter flask (see Note)]. Wash with acetone and a mixture of equal parts of acetone and chloroform until the filtrate is colourless. Digest the insoluble material, which has been carefully retained in the beaker, for 10 minutes on the steam-bath with 35 ml of sodium hydroxide solution (300 g/l). This treatment with alkali may be omitted if silicates are absent. Dilute to 60 ml with hot water and heat on the steam bath. Filter the solution of alkali and wash well with hot sodium hydroxide solution.

Note — The filtration may be materially aided, particularly with some synthetic rubber products, by partial or complete neutralization of the nitric acid solution with ammonium hydroxide. Partial neutralization together with the addition of trivalent cations or anions may also aid aglomeration of the carbon black particles, if they are too well dispersed to filter.

A-8.3.3 Next, wash the residue about four times with hot hydrochloric acid. Neutralize the last washing with ammonium hydroxide and test for the presence of lead with sodium chromate solution. If lead is present, continue to wash with hot hydrochloric acid and finally wash with warm hydrochloric acid (1:7). Remove the crucible from the funnel taking care that the outside is perfectly clean, dry it in an air-bath for 90 minutes at 110°C, cool and weigh; call this weight A. Burn off the carbon at a dull red heat (550 to 600°C) and reweigh; call this weight B. The difference in weight represents approximately 105 percent of the carbon originally present in the form of carbon black.

A-8.4 Calculation

Carbon black, percent by mass =
$$\frac{(A - B) \times 100}{1.05 \times C}$$

where

C =mass in grams of specimen used.

A-9. NITROGEN (CALCULATED AS GLUE)

A-9.1 General — This method is intended for use in the determination of glue when it is used as a filler in rubber products.

The calculations in this section are designed for determination of glue in NR products. In the absence of other nitrogenous material the method may be applied to synthetic rubber products without correction for the nitrogen content of the rubber polymer. A slight error will result from nitrogen content of antioxidants and acceleraters present after extraction. When glue is found to be present in a NR product and determined by this method by calculation from nitrogen content, a correction shall be made for the natural protein in NR as shown in the calculations. The correction shall be made by the method of

approximation. The rubber as compounded is calculated on the basis of the total nitrogen as glue. The glue content is then corrected on the assumption that the rubber as compounded contains 0.4 percent protein nitrogen and this value is used to calculate a more exact figure for rubber hydrocarbon.

A-9.2 Reagents

A-9.2.1 Copper Sulphate

A-9.2.2 Paraffin

A-9.2.3 Sodium Hydroxide (NaOH) Solution - 750 g/1.

A-9.2.4 Sodium Hydroxide Standard Solution (0.1) — Prepare and standardize a 0.1 N sulphuric acid (H₂SO₄) solution.

A-9.2.5 Sodium Sulphate

A-9.2.6 Sulphuric Acid, Standard Solution (0.1 N) — Prepare and standardize a 0.1 N sulphuric acid (H_2SO_4) solution.

A-9.2.7 Zinc Granulated

A-9.3 Procedure

A-9.3.1 Extract a 2 g specimen with acetone for 8 hours. Remove the solvent from the specimen and transfer the latter from the filter paper to a 750-ml Kjeldahl flask. Add 25 to 30 ml of sulphuric acid, 10 to 12 g of sodium hydroxide and about 1 g of copper sulphate. Heat gently until the first vigorous frothing ceases, then raise the heat gradually until the liquid boils. Continue the boiling until the solution becomes clear. Allow the flask to cool, dilute carefully with 150 ml of water and again allow to cool. Add 10 ml of sodium hydroxide solution (750 gl), pouring it carefully down the side of the flask so that it does not mix immediately with the acid solution. Add about 1 g of granulated zinc to prevent bumping and a piece of paraffin in size of a pea to diminish frothing. Connect the flask quickly with a condenser, the delivery tube of which dips into a 500-ml Erlenmeyer flask containing 50 ml of 0.1 N sulphuric acid diluted to about 100 ml. Carefully swirl the flask to mix the contents and start to heat gently, increase the flame as the danger of foaming over diminishes and finally boil briskly until about one half of the liquid has passed over into the receiver. Add methyl red solution and titrate the excess acid by means of 0.1 N sodium hydroxide solution.

A-9.4 Calculation

Total nitrogen as glue, percent =
$$\frac{(AN - BN_1) \times 0.014 \times 6.5}{C} \times 100$$

where

A =volume in millilitres of sulphuric acid used,

 \mathcal{N} = normality of sulphuric acid,

B =volume in millilitres of sodium hydroxide used,

 \mathcal{N}_1 = normality of sodium hydroxide, and

C =mass in grams of specimen used.

A-10. RUBBER POLYMER CONTENT

A-10.1 Calculation

Rubber polymer content, percent = A (100 - B)

where

A = factor as listed below:

Rubber	Factor A
NR	94/97
IR	1.00
SBR*	1.00
BR	1.00
IIR	1.00

B = sum of percentage of total extract, alcohols potash extract, organic sulphur, ash, carbon black and glue determined in accordance with A-1, A-2, A-6, A-8, and A-9 respectively.

^{*}Containing 23.5 percent bound styrene and not oil extended.

APPENDIX B

(Clause 3.1.2)

DETERMINATION OF RUBBER POLYMER CONTENT (Direct Method for Natural Rubber Composition Only)

B-0. OUTLINE OF THE METHOD

B-0.1 A weighed quantity of the material is extracted with acetone and alcohol and dried. It is then oxidized with oxidizing mixture and steam distilled. The distillate is titrated with standard sodium hydroxide.

B-1. APPARATUS

B-1.1 All connections to the flask containing the oxidizing mixture shall be through glass joints; a suitable form of apparatus as shown in Fig. 1. The apparatus shall also include a steam generator or other source of clean steam at atmospheric pressure or at not more than 15 kPa. An aeration assembly is also required consisting of a cork or bung carrying two glass tubes. This shall be fitted to the receiver after completion of the steam distillation, with the glass tubes of such length that air is removed from the top of the receiver by connecting one tube to a vacuum line, the other tube dipping well below the surface of the distillate, thus drawing air through the liquid.

B-2. REAGENTS

- **B-2.1 Chromic Acid Oxidation Mixture** Dissolve 200 g of chromic anhydride in 50 ml of water, add 150 ml of sulphuric acid (r.d. 1.84) and mix well.
- **B-2.2 Phenolphthalein** Dissolve 0.2 g of phenolphthalein in 100 ml of methylated spirit.
- B-2.3 Sodium Hydroxide 0.1 N.

B-3. PROCEDURE

- **B-3.1** Weigh accurately a sufficient amount of the sample to contain approximately 0.3 g of the rubber hydrocarbon. Extract the test portion with acetone and alcohol. Dry the extracted rubber in an oven at a temperature of 70 to 100° C for one hour.
- **B-3.2** Place sufficient water in the receiver to cover the end of the entry tube. Mark the outside of the distillation flask to indicate the liquid level when the flask contains 75 ml of the chromic acid oxidation mixture in the flask, lift the steam entry tube and insert the extracted sample. Replace the steam tube, surround the distillation flask by a boiling water

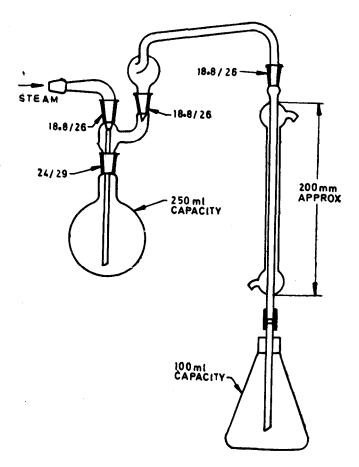


Fig. 1 Apparatus for Determination of Natural Rubber Hydrocarbon

bath and maintain boiling for one hour. At the end of this period remove the water bath and start the flow of steam, heating the distillation flask when the volume of liquid has increased to 75 ml. Adjust the heat to maintain the liquid at this level. Continue distillation until about 500 ml of liquid are in the receiver; this takes about 30 minutes. At the conclusion of the distillation remove the steam entry and stop heating. Wash the delivery tube with water into the receiver and ensure that the temperature of the liquid in the receiver is below 30°C. Connect the aeration assembly to the receiver and draw air through the liquid by means of a water

pump or other vacuum line. Maintain this stream of air for 30 minutes at approximately 2 litres per minute. At the conclusion of this time rinse the aeration assembly with water, collect the washings in the receiver and titrate the contents with 0.1 N sodium hydroxide using phenolphthalein as the indicator.

- **B-3.3** If chlorine-containing materials are present, add neutral potassium iodide to the distillate after aeration and titrate any iodine liberated with neutral sodium thiosulphate before continuing with the sodium hydroxide solution.
- **B-3.4** Make a blank determination using the same quantities of reagents and conditions of test. The sodium hydroxide titration blank should not exceed 0.3 ml.

B-4. CALCULATION

B-4.1 Rubber hydrocarbon, percent by mass = $\frac{0.908 \times V}{M}$

where

V =volume in ml of sodium hydroxide less volume in ml for blank determination, and

M =mass in g of the test portion.

B-5. INTERFERENCE BY OTHER ORGANIC SUBSTANCES

B-5.1 Mineral rubber (bitumen) is sometimes not completely removed by acetone and alcohol extraction and any residue will give some acetic acid on oxidation. The hydrocarbon in gutta percha and balata is chemically identical with that in natural rubber and returns the same values. Other polymers yield varying amounts of acetic acid on oxidation and the use of the method for a mixture of polymers should be followed by a correction for the other component, the correction being determined by oxidation of the corresponding pure material. Current samples of butadiene-styrene copolymers are equivalent to 3 percent natural rubber in the acetic acid they produce. Cellulose may be considered as equivalent to 2 percent natural rubber. Polyisobutene interferes by protecting the rubber from attack by the oxidizing mixture and for this reason the method is unreliable for determining a small amount of natural rubber mixed with a larger quantity of butyl rubber. The protein of natural rubber is not normally present in sufficient quantities markedly to interfere but in the case of a latex rubber a correction may become necessary. Four percent of protein is equivalent to 1 percent of rubber.

B-6. CORRECTION FOR COMBINED SULPHUR

B-6.1 Sulphur combined with rubber interferes in proportion to the unsaturation lost. An empirical correction is:

$$RHC = A (1 + 0.015 S)$$

where

RHC = rubber hydrocarbon,

A = apparent rubber hydrocarbon, and

S = combined sulphur as a percentage of A.

APPENDIX C

(Clause 5.2.5)

METHOD FOR THE DETERMINATION OF PERCENTAGE OF ACETONE EXTRACT AND EXTRACTABLE SULPHUR

C-1. ACETONE EXTRACT

C-1.1 Apparatus

C-1.1.1 Extraction Apparatus — The extraction apparatus is of the reflux type with the condenser placed immediately above the cup which holds the rubber. The cup is situated in the vapour of the boiling solvent and is emptied by a siphon. The apparatus is of glass except in patterns where an extraction cup is suspended from the end of the condenser, in which case platinum wire is used for the suspension. The apparatus fit together without the use of cork, rubber or metal and in such a manner that loss of vapour during extraction does not exceed 20 percent of the extracting liquid.

C-1.1.1.1 Two types of apparatus are shown in Fig. 2.

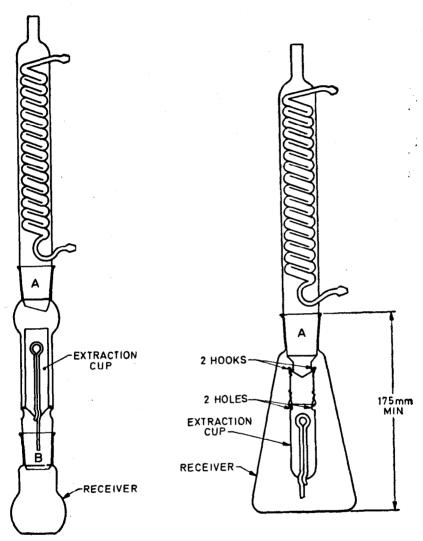
C-1.1.2 Filter Paper — acid washed hardened filament.

C-1.1.3 Nylon Cloth — open weave, continuous filament.

C-1.2 Reagent

C-1.2.1 Acetone — conforming to IS: 170-1976*.

^{*}Specification for acetone (second revision).



Type 1

Extraction	Jo	ints	Receiver	
cup	A	В		
ml 20-30 50-60	34/35 45/50	34/35 34/35	ml 150 250	

Fig. 2 Extraction Apparatus

C-1.3 Procedure — Weighed portion of the proofing (about 3 g) wrapped in a filter paper, which has previously been extracted with acetone, shall be inserted in the extraction cup and acetone poured into the extraction vessel to fill the cup about twice times. The proofing material shall then be subjected to at least 80 hot extractions during a continuous period of not less than 8 but nor more than 16 hours. After extraction, the acetone shall be distilled off from the soluble matter in a weighed vessel or preferably in the previously weighed extraction vessel itself. After the acetone has been removed, the extract shall be dried at a temperature of 70 to 75°C for one hour, cooled in a desiccator and weighed. Continue the process of drying, cooling and weighing until the change in weight between successive weighings is less than 2 mg.

C-1.4 Calculation — Calculate the weight of the extract as percentage of the proofing content as follows:

he proofing content as follows:

Acetone extract, percent by mass of the proofing content
$$= \frac{M_1}{M_2} \times 100$$

where

 $M_1 = \text{mass in grams of the extracted residue, and}$

 $M_2 =$ mass in grams of the proofing material taken for extraction.

C-2. EXTRACTABLE SULPHUR

C-2.1 The acetone extract of the sample, obtained as described in B-1.3, shall be oxidized by bromine in the presence of water. From the resulting sulphuric acid, the amount of sulphur shall be estimated, gravimetrically as barium sulphate and expressed as a percentage of the weight of proofing content.

APPENDIX D

(Table 1)

DETERMINATION OF PROOFING CONTENT

D-0. OUTLINE OF THE METHOD

D-0.1 Test pieces of known area and mass after conditioning are refluxed in a suitable solvent to remove polymer from the fabric.

D-1. REAGENT

D-1.1 Solvent Capable of Swelling or Dissolving Cured Polymer Present in the Sample — The choice of a suitable solvent depends upon the nature of the polymer and upon the properties of the textile base.

D-2. PROCEDURE

D-2.1 Cut 4 test pieces each approximately 10×10 cm equally spaced across the width of the fabric with the 2 test pieces centered 15 cm from the selvedges. After conditioning at $27 \pm 2^{\circ}$ C and 65 ± 2 bercent relative humidity immerse each test piece in the solvent and heat under the reflux until the proofing is dissolved or swollen thoroughly (generally about one hour) taking care not to allow the temperature to exceed 160° C. Remove the test piece from the solvent and carefully scrape off any swollen proofing with a spatula. Repeat the treatment using fresh solvent on each occasion until the fabric is free from rubber. Squeeze the fabric to remove solvent and rinse with light petroleum hydrocarbon solvent until free from the original solvent. Dry for one hour at 105 to 110° C and then condition for 24 hours. Transfer to a weighing bottle and determine the weight of the fabric.

D-2 2 Ash the fabric at a low temperature in a previously ignited weighed silica crucible and determine the weight of the ash.

D-3. EXPRESSION OF RESULTS

D-3.1 The mass of proofing, grams per square metre (as determined on 10×10 cm test piece) = 100 (a - b + c)

where

a =mass in g of the original test piece,

b =mass in g of the fabric after removal of proofing, and

c =mass in g of ash of the fabric.

APPENDIX E

(Clause 5.3.2)

DETERMINATION OF WATERPROOFNESS

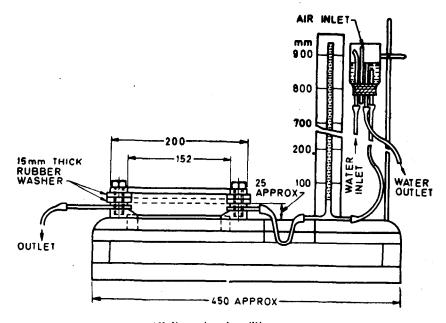
E-1. TEST SPECIMENS

E-1.1 The test specimens shall be circular pieces, each 20 cm in diameter, cut from the sample.

E-2. APPARATUS

E-2.1 Any type of apparatus which can subject the rubberized fabric over a circular portion, 15 cm in diameter to the specified water head for

a specified period may be used. A suitable form of apparatus (see Fig. 3) consists of a trough and ring, the opposing faces of which are machined and both are drilled at 8 corresponding places to take 8 bolts. Two rubber washers are fitted between the trough and the ring. The trough is provided with an exit valve and is connected to pressure gauge and device for adjusting the water head and maintaining it at a constant value. For convenience the trough is mounted on a hinged base so that it can be swung through a right angle to permit release of the entrapped air.



All dimensions in millimetres.

FIG. 3 WATERPROOFNESS TEST - LOW PRESSURE

E-3. PROCEDURE

E-3.1 The test piece shall be fitted into the apparatus, with a rubber washer on each side and assembly clamped tightly together. The pressure of water head shall gradually be raised to 90 cm using water at room temperature. Under this pressure the material shall be kept for one hour. Examine the fabric for any leakage of water and wetting.

E-4. RESULT

E-4.1 The material shall be declared satisfactory if there is no leakage of water through the fabric and wetting of outer surface of the fabric.

APPENDIX F

(Clause 7.1)

SAMPLING OF DOUBLE-TEXTILE RUBBERIZED WATERPROOF FABRICS

F-1. SCALE OF SAMPLING

- **F-1.1 Lot** In a consignment, all the rolls of same quality, same grade and belonging to the same batch of manufacture shall be grouped together and each such group shall constitute a lot.
- F-1.2 For ascertaining the conformity of material to the requirements of the specification, samples shall be tested from each lot separately.
- F-1.3 The number of rolls to be selected from a lot shall depend upon the size of the lot and shall be according to Table 2.

TABLE 2 SCALE OF SAMPLING

NUMBER OF ROLLS IN THE LOT	Sample Size
(1)	(2)
Up to 50	2
51 to 100	3
101 to 150	4
151 to 300	5
301 and above	7

F-1.3.1 The rolls shall be selected from the lot at random. In order to ensure the randomness of selection a random number table shall be used. For guidance and use of random number tables, IS: 4905-1968* may be referred. In the absence of the random number table, the following procedure may be adopted:

'Starting from any roll in the lot, count them in order as $1, 2, 3 \dots$ etc, up to r and so on where r is the integral part of N/n (N being the number of rolls in the lot and n the number of rolls to be selected from the lot). Every rth roll thus counted shall be withdrawn till the requisite number of rolls is obtained.'

^{*}Methods for random sampling.

F-2. TEST SAMPLES AND REFEREE SAMPLES

F-2.1 From each of the rolls selected according to **F-1.3**, a specified length of fabric shall be cut, care being taken to exclude not less than 25 cm length of fabric from either end. The length shall be adequate to provide test specimens to be cut from this length for various tests given in the specification.

F-3. NUMBER OF TESTS AND CRITERIA FOR CONFORMITY

- F-3.1 The test for all the requirements given in the specification shall be conducted on individual samples.
- **F-3.2** The lot shall be declared as conforming to the requirements of the specification if for each of the characteristics all the test results on individual samples are found to be meeting the corresponding specification limits.

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Inks and allied products	Thinners and solve
Laboratory glassware thermometers	Varnishes and lacq
and related apparatus	Water and water tr
Lac and lac products	
Leather, leather goods and leather	Water based paint

dressings

Fillfals allo sillen bionners
Lubricants and related products
Oil pastes
Oils and fats, oleaginous seeds and
fruits
Painters' materials (miscellaneous)
Paper and its products
Paper and pulp board packaging
materials
Perfumery materials, natural and
synthetic
Petroleum and petroleum products
Photographic chemicals
Pigments and extenders
Plastics
Polishes
Printing inks
Ready mixed paints and enamels
Rubber and rubber products
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agents
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AMENDMENT NO. 1 JUNE 1986

TO

IS: 6110-1933 SPECIFICATION FOR DOUBLE-TEXTURE RUBBERISEL WATERPROOF FABRICS

(First Ravision)

(Page 6, clause 5.2.4, last paragraph) - Substitute the following for the existing paragraph:

'Four test pieces, two in the warp direction, and two in the west direction shall be subjected to this test in original condition. A similar set of test pieces shall be subjected to this test after accelerated ageing.

АНЕНДИЕНТ NO. 2 МАУ 1990 ТО

IS 6110:1983 SPECIFICATION FOR DOUBLE-TEXTURE RUBBERISED WATERFROOF FABRICS

(FOURTH REVISION)

(Page 5, clause 5.3.2, last line) Substitute 'when tested in accordance with method A-1 of IS 7016 (Part 7): 1986*' for 'when tested In accordance with Appendix E'.

(Page 27, Appendix E) - Delete and renumber nubsequent appendices accordingly.

* Nethodo of tent for coated and treated labilitate Part 7 Determination of resistance to penetration by water (first revision).

(PCD 16)

AMENDMENT NO. 3 MAY 2007 TO IS 6110: 1983 SPECIFICATION FOR DOUBLE-TEXTURE RUBBERIZED WATERPROOF FABRICS

(First Revision)

(Page 19, clause A-9.2.4) — Substitute the following for the existing:

'Prepare and standardize a 0.1 N sodium hydroxide (NaOH) solution.'

(PCD 13)